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13. ABSTRACT (Maximum 200 words)

In cement-based materials, cement is usually employed to bind other materials together. Different interfaces are thus generated between various media in the resulted materials. It has been well recognized that the interfacial microstructure between cement binder and inclusions is of the greatest importance for the mechanical properties of the composite. Specifically, in fiber reinforced cementitious composites, the composite material properties are especially predominated by the fiber-cement interface due to its influence on the mechanical interactions between fibers and cement matrix. This report summarizes research investigations and findings of a study of the fiber-cement interfacial debond mode, namely strength-based or fracture-based debond modes, and on an issue of interfacial bond property control. These studies have been done with the help of fiber pull-out experiments using an MTS digitally controlled load frame and micromechanical modeling, accompanied by environmental scanning electron microscopy. We expect that such investigations will provide physical insights into the break-down processess occurring at the interphase levels of fiber reinforced cementitious composites. The knowledge, in turn, many serve to achieve fiber reinforced cementitious composites with higher performance.

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MECHANICAL INTERACTIONS BETWEEN SYNTHETIC FIBER AND CEMENT BASED MATRIX IN FRC COMPOSITES

Final Technical Report
February, 1993

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[1] Disclaimer: The views and conclusions contained in this document are those of the authors and should not be interpreted as necessarily representing the official policies, either expressed or implied, of the United States Government.

Table of Content

Introduction

In cement-based materials, such as mortar, concrete, steel reinforced concrete, fiber reinforced concrete, etc., cement is usually employed to bind other materials together. Different interfaces are thus generated between various media in the resulted materials. It has been well recognized[1] that the interfacial microstructure between cement binder and inclusions is of the greatest importance for the mechanical properties of the composite. Specifically, in fiber reinforced cementitious composites, the composite material properties are especially predominated by the fiber-cement interface due to its influence on the mechanical interactions between fibers and cement matrix.

This is a report which provides some details of the research investigations and findings of the period since the last report date. This report also summarizes the major findings of the complete research project, and as such, constitute the final project report to AFOSR.

Project Summary

During the last investigation period, we have focused on a study of the fiber-cement interfacial debond mode, namely strength-based or fracture-based debond modes, and on an issue of interfacial bond property control. These studies have been done with the help of fiber pull-out experiments using an MTS digitally controlled load frame and micromechanical modeling, accompanied by environmental scanning electron microscopy. Works on these aspects are described further below, and the findings are reported. We expect that such investigations will provide physical insights into the break-

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[1] Mindess, S. (1988) "Bonding in Cementitious Composites: How important is it?" *Mat. Res. Symp. Proc.* V. 114, p. 3-10.

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down processes occurring at the interphase levels of fiber reinforced cementitious composites. The knowledge, in turn, may serve to achieve fiber reinforced cementitious composites with higher performance.

Investigations

Researches conducted in the last investigation period was aimed at improving our basic understanding of the micromechanisms associated with interfacial debonding and pull-out of fibers embedded in a cementitious matrix. The objective is to make possible the control of the interfacial bond properties based on the knowledge obtained from these investigations. The technology of bond property control, in turn, will be applied in composite materials design to achieve the desired material properties.

(1)   Interfacial debond mode

In general, the interfacial bond properties refer to the interfacial bond strength or the critical interfacial energy release rate, which controls the onset of interfacial debonding, and the frictional resistance between fibers and adjacent cement material during the pull-out process. In elastically bonded region, whether the initiation of interfacial debonding is determined by the interfacial bond strength or the critical interfacial energy release rate depends on the components of the composite materials. While the interfacial debond mode is a material characteristic, most of the micromechanical models for fiber reinforced cementitious composites have been developed based either on strength criterion or on fracture criterion. Erroneous assumption of the debond mode will lead to the accessment of interfacial properties which have no real physical meaning. It is crucial to employ the right debonding criterion for the right material system. Therefore, the

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determination of interfacial debond mode for any particular material system is important in order to adopt the appropriate debond criterion.

It has been found[23] that both strength-based and fracture-based debonding criteria may result in similar fiber stress versus pull-out displacement, σ_p-u, relations. In particular, the expressions for σ_p-u relations from both approaches can be exactly the same when an effective bond strength is introduced.

$$\tau_{eff} = \begin{cases} \tau_s & (\text{strength} - \text{based approach}) \\ \tau_i + \sqrt{\dfrac{2E_f\Gamma(1-\alpha)\rho^2}{d_f}} & (\text{fracture} - \text{based approach}) \end{cases}$$

in which

τ_{eff}:   effective interfacial bond strength,

τ_s: elastic interfacial bond strength,

τ_i: frictional stress,

Γ: critical interfacial energy release rate,

E_f: modulus of elasticity of fiber,

d_f: fiber diameter,

$$\alpha = \frac{E_f V_f}{E_c}$$

$$\rho^2 = \frac{2G_m E_c}{V_m E_m E_f \log\left(\dfrac{R^*}{r_f}\right)}$$

where

E_m and E_c: modulus of elasticity of matrix and composite,

G_m: matrix shear modulus,

[2]Leung, C.K.Y. and Li, V.C. (1990a). "Strength-Based and Fracture-Based Approaches in the Analysis of Fiber Debonding." *J. Mat. Sci. Letters*, 9, p.1140-1142.
[3]Leung, C.K.Y. (1992). "A Fracture-Based Two-Way Debonding Model for Discontinuous Fibers in an Elastic Matrix." *ASCE J. Eng. Mech.*, 118(11), p.2298-2318.

V_m: matrix volume fraction, i.e., $1 - V_f$, and

r_f: fiber radius.

R^* is the radius of a matrix cylinder containing the fiber within which the matrix is assumed to deform in shear only and all axial stress is localized on the cylinder rim at R^* [4].

The similarity between the solutions from the two approaches makes it impossible to determine whether debonding is strength governed or fracture governed if interfacial bond properties is measured with a single type of specimen. Nevertheless, the dependency of τ_{eff} on d_f for the fracture models and the independency of τ_{eff} on d_f for the strength models suggest a simple approach to identify the true mode of interfacial debonding. While both τ_s and Γ can be derived from a given set of fiber pull-out test result, only one of these two parameters has physical meaning. If a fiber-matrix material system follows strength-based debonding criterion, the interfacial bond strength measurement, via single fiber pull-out test for instance, shall not be affected by the fiber sizes employed as long as the fibers of various diameters are made by the same material with the same surface treatment. On the other hand, for material systems following fracture-based debonding criterion, the interfacial critical energy release rate or fracture toughness would be the true material parameter with respect to interphasal debonding. In this case, the measured Γ's have to be constant regardless of various fiber sizes.

An experimental method for determining the debonding mode has been proposed based on the concept described above. Single fiber pull-out tests were conducted for the material systems with various fiber diameters. τ_{eff} and τ_i were then determined by the procedure

[4] Budiansky, B., Hutchinson, J.W. and Evans, A.G. (1986). "Matrix Fracture in Fiber-Reinforced Ceramics." *J. Mech. Phys. Solids*, 34(2), p.167-189

described in Li and Chan (1993)[5]. Dependency of the resulting τ_{eff} on d_f was then examined in an attempt to identify the interfacial debond mode.

(2)   The characteristics of interfacial debonding

In previous section, it has been pointed out that the "effective bond strength" may be employed to substitute both elastic bond strength and the interfacial critical energy release rate regardless the real debond mode. In order to quantitatively assess the bond properties in various conditions and to develop the methodology of bond property control, it is decided to employ the effective bond strength, or simply the "bond strength", to represent the general bond properties in the following sections.

For the purpose of bond property control, the characteristics of interfacial debonding were investigated. Due to the uniqueness of the microstructure of cementitious material, there are several potential weak phases responsible for the mechanism of fiber-cement interfacial debonding. Specifically, the interface between fibers and adjacent cement material as well as the porous interphasal zone can be the weak phase where interfacial debonding eventually occurs.

Due to the porous nature of the interphasal zone between the fiber and the matrix of a cementitious composite, it is expected that densification in microstructure may serve to strengthen this interphasal zone and increase the interphasal bond strength. However, for fibers made up of materials with low surface energy, such as polymeric fibers, the adhesive strength between fiber and matrix may be the controlling parameter for fiber-matrix bond strength. For such materials, densification of interphasal zone may not be effective in bond improvement. For such material systems, appropriate fiber surface

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[5]Li, V.C. and Chan, Y.W. (1992). "Determination of Interfacial Debond Mode for Fiber Reinforced Cementitious Composites." Submitted for publication in *ASCE J. Eng. Mech.*.

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modification may be required to enhance the adhesive strength between fiber and cement matrix. The assessment of the characteristic of interfacial debonding, namely debonding occurs at the fiber-cement interface due to a low adhesive strength or in the relatively porous interphasal zone, becomes very important in order to effectively improve the interfacial bond properties.

In this regard, the effect of interphase densification on the improvement of the bond properties was investigated. An attempt was made to densify the fiber-matrix interphasal zone of a steel-cement system so as to enhance the corresponding bond strength. Several methods were attempted, including reduction in w/c ratio and incorporation of silica fume. Measurements of bond strengths based on single fiber pull-out tests were carried out. The investigation was conducted using both steel and brass fibers. With the contrast between steel-cement and brass-cement systems, the importance of the chemicophysical properties of fiber surface is then addressed. A toughening mechanism associated with the bond property was established based on the linkage between the microstructural morphology as observed by Environmental Scanning Electron Microscope (E-SEM) technique and the corresponding interfacial bond property determination.

It was found that the debonding locations, at the fiber-cement interface or in the interphasal zone, is dependent on the adhesive strength between fiber and matrix as well as the cohesive strength of the interphasal zone. This leads to different strategies for bond property control. For material systems having relatively lower cohesive strength of the interphasal zone, bond failure may occur in the interphasal zone and the densification of interphasal zone can enhance the bond properties by strengthening the cohesive strength of the interphasal zone. On the other hand, there are material systems with relatively lower adhesive strength between fiber and matrix, the debonding may thus initiate at the fiber-cement interface. In order to enhance the bond properties, fibers may require

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appropriate surface modification in order to increase the adhesion strength and, secondly, the interphase densification may further improve the bond properties.

Experimental Program

1. Determination of Interfacial Bond Properties

Fiber pull-out tests were conducted in the test program by pulling individual fibers out of cement matrix. The single fiber pull-out test setup is shown in Figure 1. Basically, a fiber sample is partially embedding in the dog-bone shape specimen and partially protruding. A hydraulic grip, which is subjected to a constant displacement rate, is adopted to hold the fiber such that no slip between the grip and the fiber may occur. The pull-out load is obtained from the load cell through a data acquisition system. The corresponding displacement of the fiber protruded end is recorded simultaneously. In practice, the displacement of the fiber protruded end is obtained by subtracting the elastic stretch of the fiber free length from the relative displacement between the matrix base and the hydraulic grip. This relative displacement is measured using a clip-gage. The elastic stretch of the fiber free length between the matrix base and the hydraulic grip at any given load is calculated based on the initial fiber free length, fiber cross-sectional area, and fiber elastic modulus.

Specimens were demolded 24 hours after casting and were cured in a water tank till testing. At least 6 specimens were tested for each case. Pull-out tests were conducted under a uniaxial hydraulic MTS testing machine.

2. Fiber-Cement Interphase Microstructural Observation

Morphologies of microstructures of the cement matrices as well as the debonded surfaces on both matrix side and fiber side were observed by ElectroScan environmental scanning electron microscope (E-SEM). Matrix microstructures were obtained by observing the fracture surface of each cement matrix. The debonded surfaces were created by peeling off the embedment fiber from the matrix, since the essential features of the debonded surfaces are likely to be destroyed if subjected to a pull-out process.

3. Material Systems

The mix proportions of cement matrix of various packing density are given in Table I.

Table I   Compositions of Cement Matrix

Cement Matrix	w/c	SF/c [1]	SP/c [2]
I	0.4	--	--
II	0.27	--	1.2%
III	0.27	20%	3%

[1] Silica fume to cement ratio by weight.
[2] Superplasticizer to cement ratio by weight.

The fibers employed in the bond property measurement include steel, brass, polyethylene (Spectra), and polypropylene fibers. Various fiber diameters are summarized in Table II.

Table II   Diameters (in mm) for each Fiber Type

Steel	Brass	Polyethylene	Polypropylene
0.38	-	(38 μm)	0.8
0.51	0.51		
0.81	0.81		
1.02	1.02		

Results and Discussions

1.   For the tested material systems, the debond mode has been found to be strength-based.

The tested material systems, steel-cement and brass-cement, are generally frictional control, therefore the bond strengths are identical to the frictional stresses. The interpreted bond properties are summarized in Figure 2 and 3. For each curing age, steel-cement bond strengths for different fiber diameters are comparable and no significant dependency on fiber diameter can be found as shown in Figure 2. In Figure 3, the brass-cement bond strength is also independent of fiber diameters. According to the criteria of debond mode discussed previously, the interfacial debonding of these system may therefore be identified as strength-based.

2. **Interphase densification can be achieved by using low w/c ratio or introducing micro silica as additive.**

In the investigation of the effect of interphase densification, different cement mixes with various packing densities were employed. According to SEM observations, it is clearly indicated by the microstructural evidence that the packing density of the cement matrix can be increased by either using lower w/c ratio or introducing condensed silica fume. The morphologies of the microstructure of the interphasal zone of Matrix I-III are shown in Figure 4(a)-4(c) correspondingly.

3. **For steel-cement system, the weak phase where debonding occurs is at the fiber-cement interface. Densifying the interphasal zone does not provide improvement to the corresponding bond properties.**

The influence of cement matrix density on the interphasal bond strength of steel-cement was found to be negligible. For various w/c ratios, with and without silica fume, the steel-cement bond strengths are in the order of 4 MPa with a slight deviation as summarized in Figure 5. On the other hand, the bond strengths between brass fiber and cement matrix are significantly affected by matrix packing density. The brass-cement bond strength obviously increases with the matrix packing density. Microstructural observations on tested and untested fiber samples, given in Figures 6 and 7, indicate that interfacial bond failures of the two systems, steel-cement and brass-cement, occur in different locations: the steel-cement interface and the interfacial zone between brass fiber and cement matrix. With the add of microstructural evidence, it is inferred that steel-cement system, having lower adhesive strength and tending to debond at the fiber-cement

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interface, does not benefit from interphase densification. In brass-cement system, the weak phase being the interphasal zone. the densification of interphasal zone can really improve the bond properties.

4. **The strategy for bond property improvement suggests appropriate fiber surface modification is required for those material system with low adhesive strength between fiber and matrix, which is especially true for synthetic fibers. Typical example is given by the Plasma-treated Spectra fiber reinforced composites.**

Interfacial bond strengths of different fibers are summarized in Figure 8, including brass, steel, polypropylene, and polyethylene (Spectra) fibers. It is clear that polymeric fibers, such as polypropylene and polyethylene fibers, have much lower interfacial bond strength with cement matrix due their inertness and low surface energy. Bond strength of steel fiber, although higher than those of polymeric fibers, due to its inertness to cement material, is slightly lower than that of brass fiber. As described previously, while steel-cement debonds at the fiber-matrix interface, brass-cement debonds in the interphasal zone. According to Figure 8, it is suggested that the cohesive strength of the interphasal zone can be in the order of 4 MPa. Material systems with adhesive strength between fibers and cement matrix lower than this margin value may not benefit from interphase densification as mentioned in 2. In order to enhance the bond properties effectively, appropriate fiber surface modification is required. For instance, Spectra fibers after Plasma treatment may increase their surface reactivity and result in a better bond strength with cement matrix than fibers without treatment. As shown in Figure 9, the treated Spectra fiber reinforced cementitious material almost doubles the maximum bridging stress and ultimate strain. This

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demonstrates that appropriate surface treatment can actually improve the surface conditions of fibers to develop better bond properties when used in cementitious composites.

Final Project Conclusions

1. For the cases of stable debonding, such as most synthetic fibers, fiber debonding process initiates gradually from the protruded end before reaching the peak load. This phenomenon is consistent with the theoretical prediction given by shear lag model.

2. For synthetic fibers, the frictional stress in the pull-out process can be increased due to the abrasion effect. This phenomenon is especially significant when fibers are not straight but in a curvy shape. The enhancement in the frictional stress was found to increase with the fiber curvature.

3. In the material systems tested, steel-cement and brass-cement, the obtained bond strengths indicate that the appropriate debonding criterion is strength-based. Furthermore, the frictional stress is found to be the dominant bond properties, a single parameter τ_i would be adequate to describe the interfacial debonding in a composite model for these particular material systems. The interfacial debond mode, strength-based or fracture-based, is material property and is dependent of the components of composite. While the material systems tested in this program are categorized to be strength-based, the suitable debonding assumption for fiber

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reinforced composite materials made up of different fibers and matrices needs to be identified for different material systems.

4. Interphasal debonding was found to occur either at the fiber-cement interface or at the heterogeneous interphasal zone, depending on the adhesion between fiber and cement and on the strength of the interphasal zone. Different debonding mechanism leads to different strategy of controlling fiber-cement bond strength. For steel fiber and synthetic fibers, such as polyethylene (Spectra) and polypropylene fibers, increasing the cement density or compactness may not necessarily improve the fiber-cement bond strength because of the lower adhesive strength. It is concluded that simultaneous applications of interphase densification (e.g., by refining microfiller grading) as well as fiber surface modification (e.g., by Plasma treatment) may lead to much more desirable bond properties.

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Tension-softening Relation in Fiber Reinforced High Strength
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1st Bolomey Workshop on Numerical Models and Material Parameters
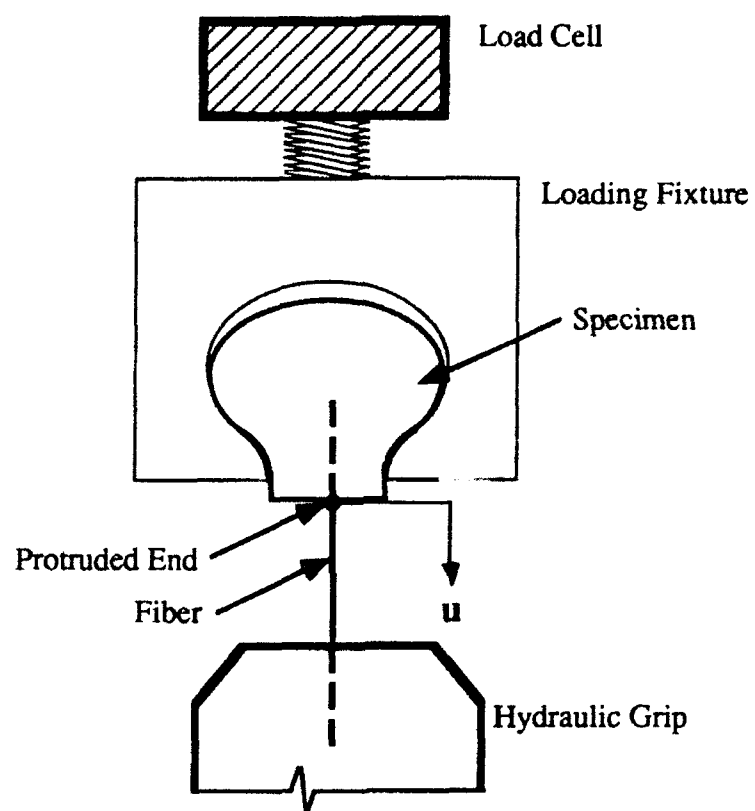for Concrete Cracking, Ed. F. Wittman, pp. 33 - 43, 1992.

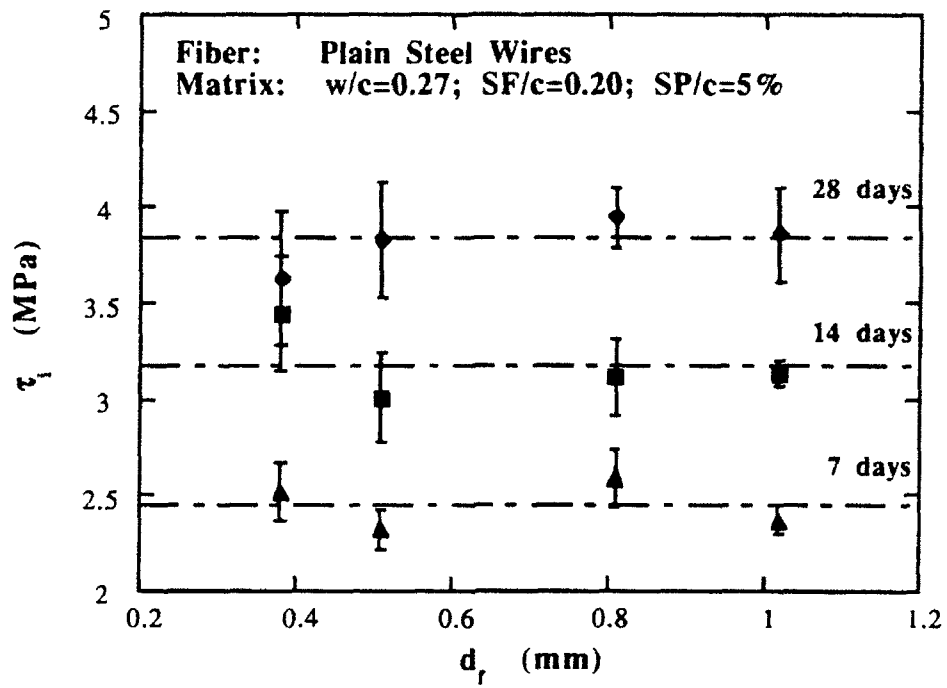**Figure   1   Single Fiber Pull-out Test Setup**

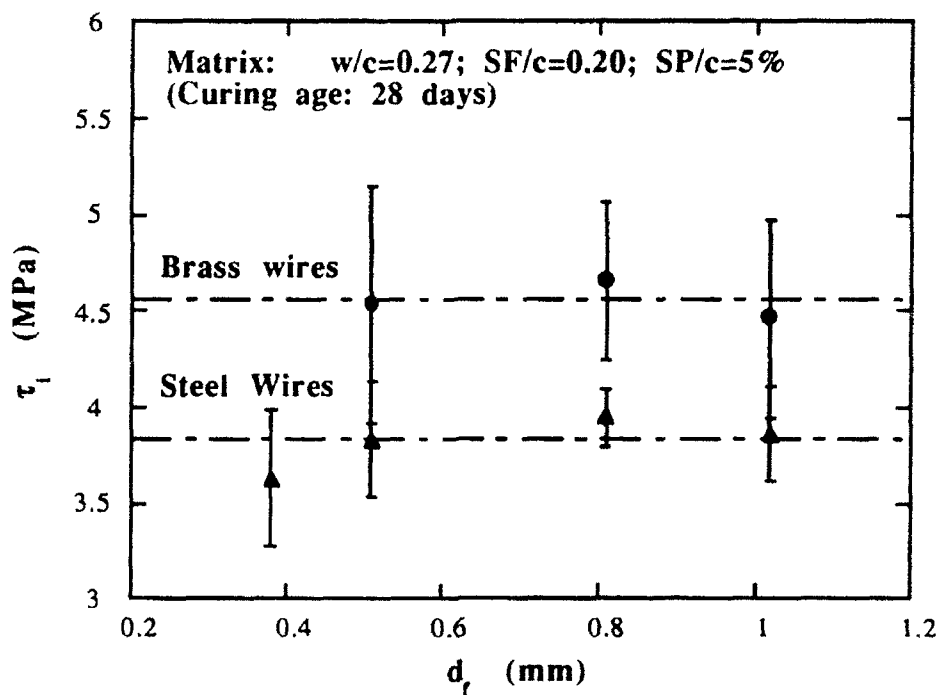**Figure   2   Steel-Cement Bond Strength Determined from Pull-out Test using Four Fibers with Different Diameters**

Figure   3   Comparison of Bond Strength-Fiber Diameter Relation between Steel-Cement and Brass-Cement Systems

Figure   4(a)   Microstructure of Matrix I (w/c=0.40)

Figure   4(b)   Microstructure of Matrix II (w/c=0.27)

Figure   4(c)   Microstructure of Matrix III (w/c=0.27 and SF/c=0.20)

4 (a)
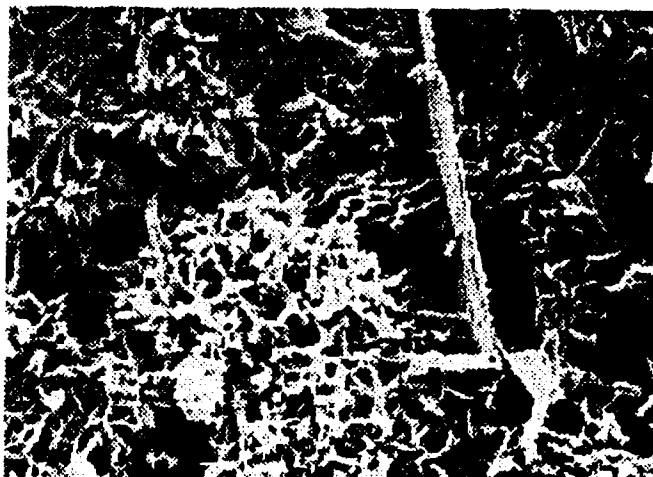
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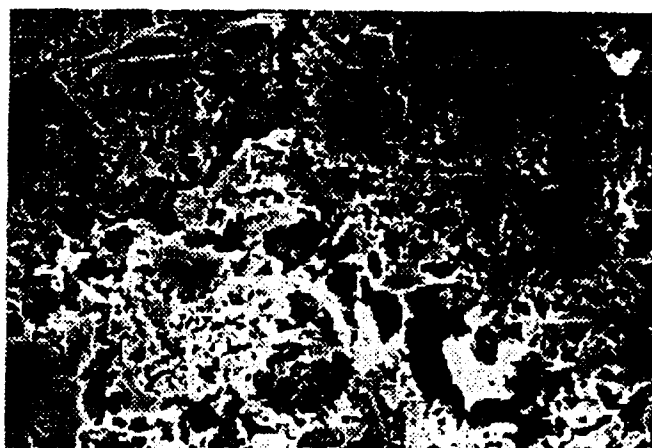
4 (c)
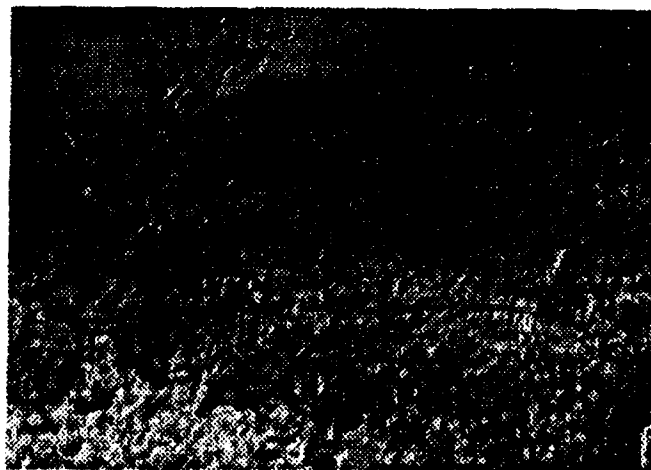
Figure 4 Microstructure of Matrix I ($w/c=0.40$), II ($w/c=0.27$), and III ($w/c=0.27$, $SF/c=0.20$)

Bond Strength of Steel and Brass Fibers with Various Matrix
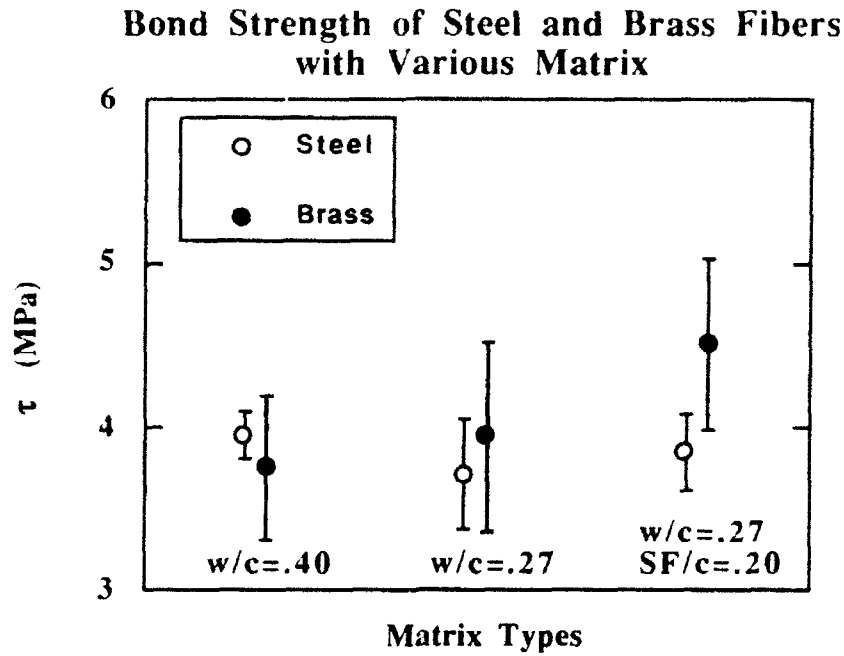
Figure 5 — Steel and brass bond strengths with respect to various cement matrices

Figure 6(a) — Groove surface on the cement matrix after the fiber peeled off (steel fiber-Matrix III)

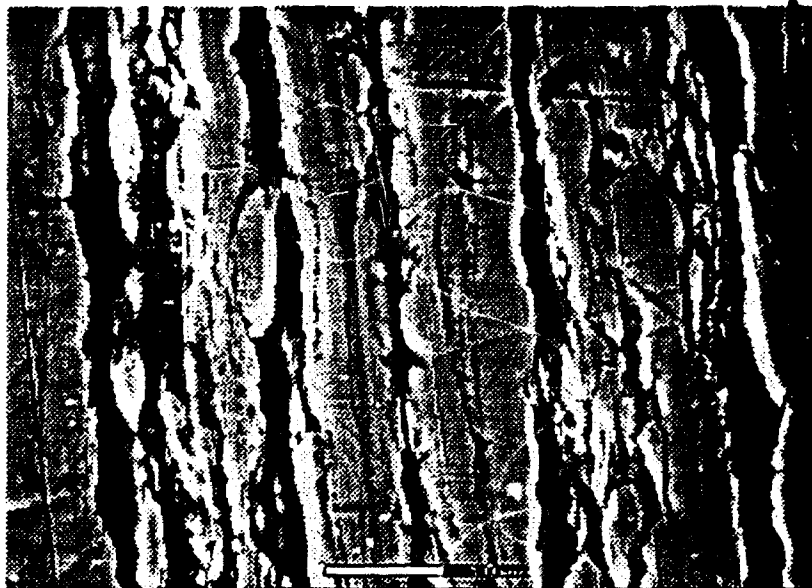Figure 6(b) — Steel fiber surface after separation from the cement matrix (Matrix III)

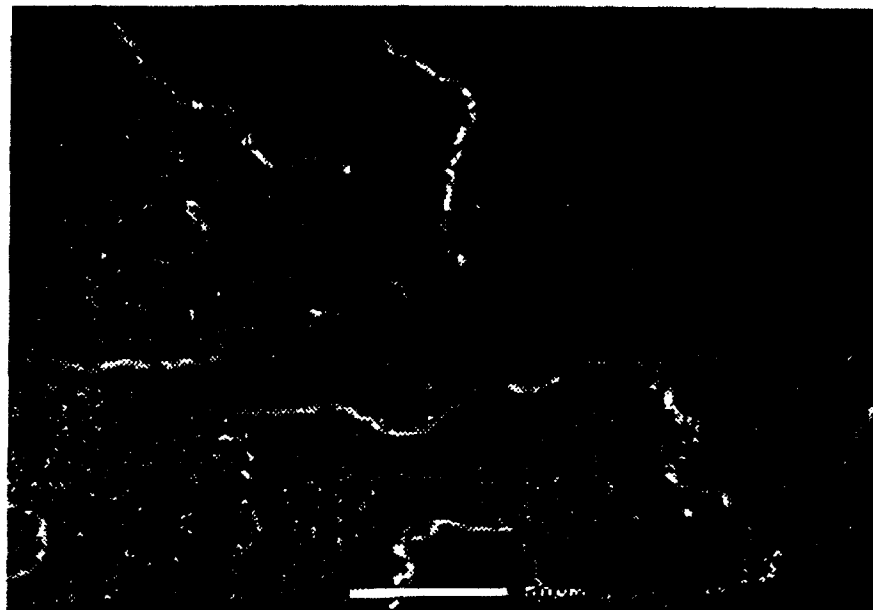Figure 7(a) — Groove surface on the cement matrix after the fiber peeled off (brass fiber-Matrix III)

Figure 7(b) — Brass fiber surface after separation from the cement matrix (Matrix III)

6 (a)
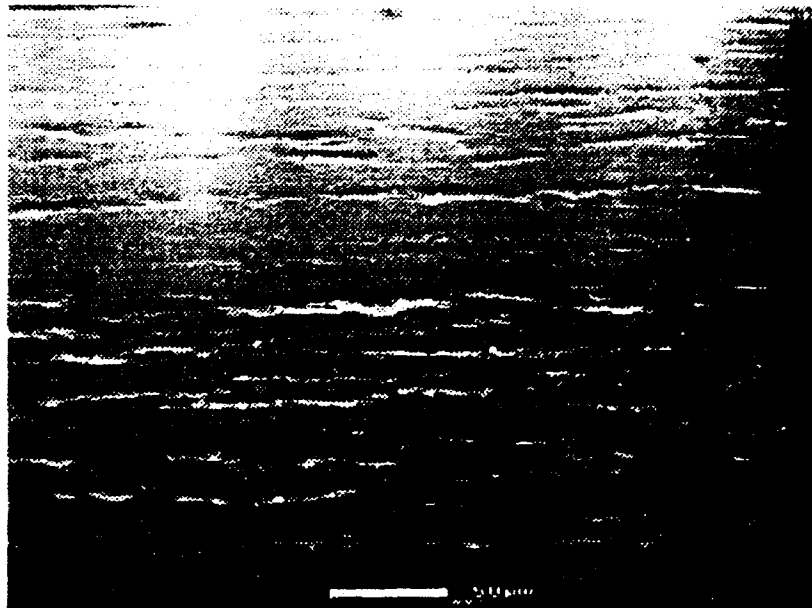
6 (b)

Figure 6   Groove surface and fiber surface after steel fiber peeled
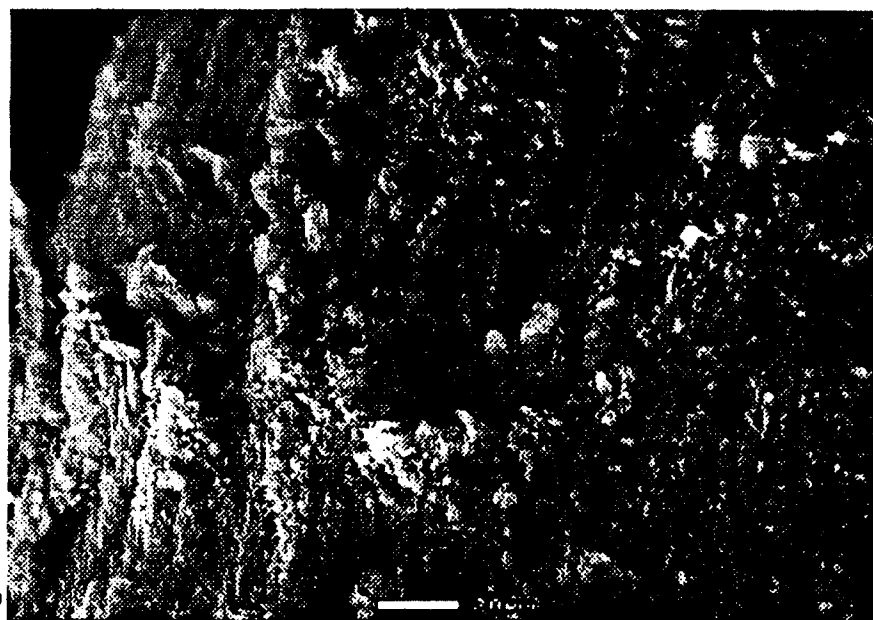off from matrix. (Matrix III)

7 (a)

7 (b)

Figure 7 Groove surface and fiber surface after brass fiber peeled
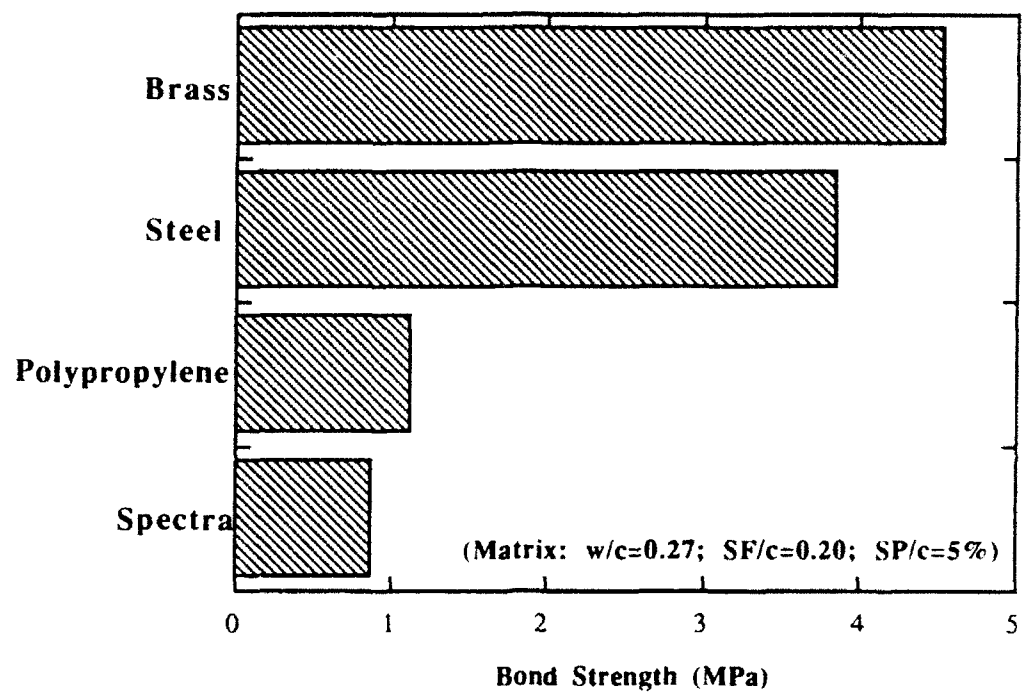off from matrix. (Matrix III)

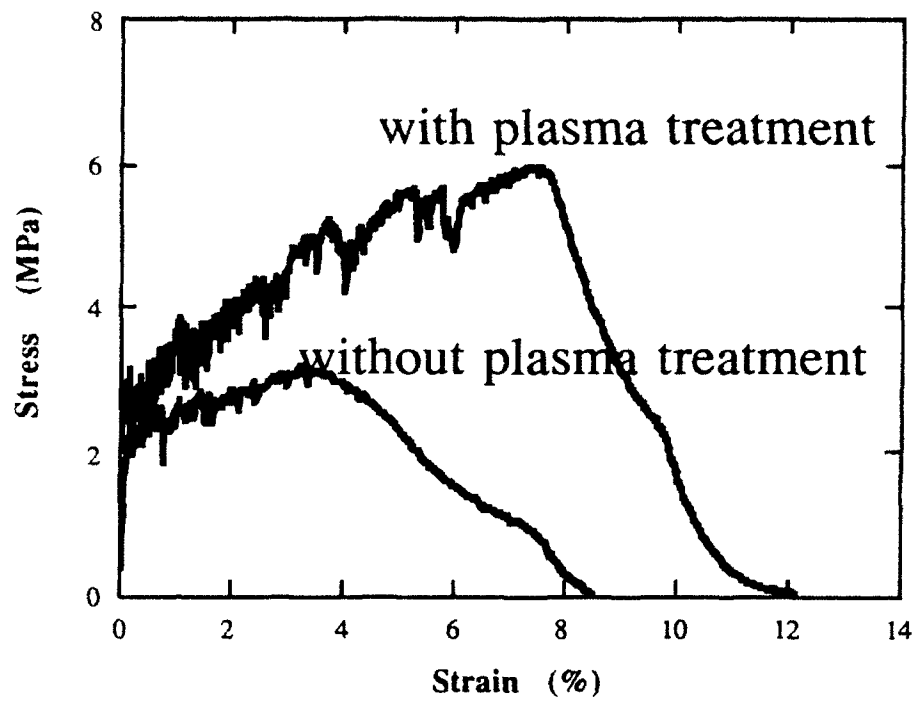**Figure   8   Typical bond strength of different fibers**

Figure 9 Effect of Plasma treatment on mechanical properties of Spectra fiber reinforced composite